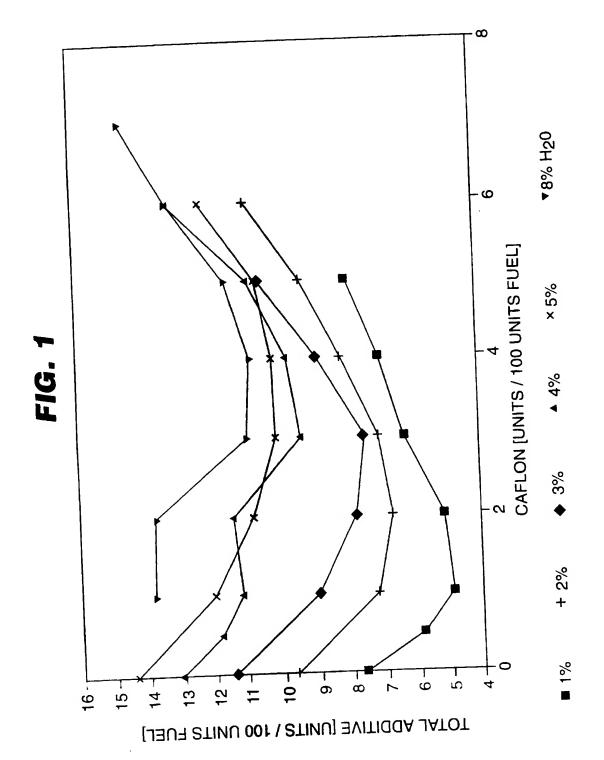
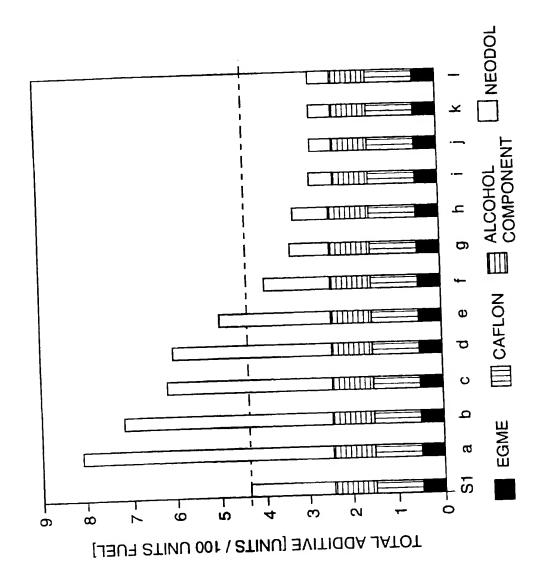
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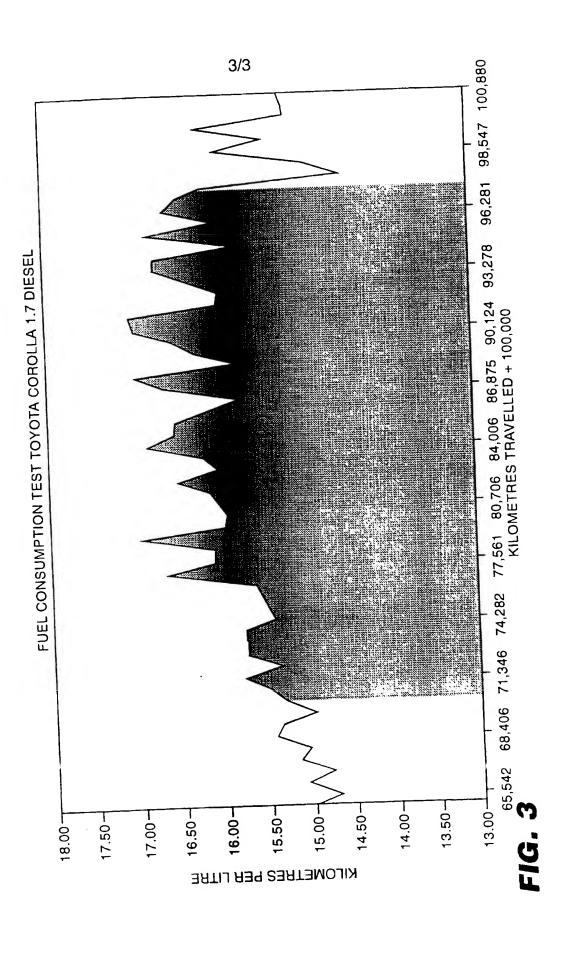
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- (51) INT CL6 (21) Application No 9807792.8 B01F 17/22 , C10L 1/18 1/22 (22) Date of Filing 09.04.1998 (52) UK CL (Edition Q) **B1V** VB VE V107 V108 V109 V205 **C5G** GAA G121 (71) Applicant(s) Coval Technologies Limited (56) Documents Cited (Incorporated in the United Kingdom) WO 98/17745 A1 41 Pound Road, East Peckham, Tonbridge, Kent, GB 2217229 A Chemical Abstract No. 95:135609 & DE 002940782 A TN12 5AY, United Kingdom (58) Field of Search UK CL (Edition P) B1V VB VE VF , C5G GAA GAB (72) Inventor(s) Peter Street INT CL6 B01F 17/00 17/22 , C10L 1/18 1/22 Albert Reginald David Thorley Online: WPI, Claims, Japio, CAS (74) Agent and/or Address for Service Carpmaels & Ransford 43 Bloomsbury Square, LONDON, WC1A 2RA, United Kingdom
 - (54) Abstract Title Solubilising water and fuel oil
 - (57) A solubilising composition which allows fuel oil and water, especially diesel oil and water, to be combined as a clear, stable homogenous solution comprises (a) an alcohol ethoxylate having a hydroxyl number greater than 160 and melting point below - 150C; and (b) a complex mixture of higher fatty acid diethanolamides derived from coconut oil, or its synthetic equivalent. The composition may also contain minor proportions of (c) ethylene glycol monobutyl ether or monobutyl ester or (d) an alcohol containing 5 or more carbon atoms. Fuel extenders such as ethanol may also be included in the compositions.







SOLUBILISING COMPOSITION

This invention relates to a composition that enables water and fuel oil to be combined into a mixture which behaves as a clear stable homogeneous solution. The composition may be used, for example, in forming stable solutions of higher fraction light fuel oils and gas oils and water, and finds particular use as an additive to diesel fuel oil that has previously been contaminated with water or to diesel fuel oil that might be expected subsequently to suffer from such contamination.

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The presence of water in fuel oil can cause a number of difficulties. If the water is present as an undesired contaminant, the burning of the resultant fuel is often uneven or, if the fuel is used in an engine, erratic running and stalling often results together with a serious risk of corrosion and possibly mechanical damage. In addition, the presence of water in fuel oils in amounts too small to give rise to such difficulties can still be undesirable as it can produce a lack of clarity or haze which is unappealing to the consumer. On the other hand, there are occasions when it might be desirable to be able deliberately to blend water with fuel oils in a stable mixture to act an extender. Alternatively such a deliberate blend of water with fuel oils may assist in the blending of other extenders (e.g. alcohols, vegetable oils etc.) for the known improvements in performance and cleaner exhaust emission that can result in, for example, engines or furnaces using diesel fuel or gas oils.

A large number of attempts have been made to deal with these problems by attempting to create stable emulsions of diesel or other fuel oil with water. Such attempts, even with proprietary brands of additive (diesel fuel improvers), have not been wholly successful because long-term stability of the resulting mix has not been achieved in practice. As the emulsions or suspensions formed with the addition of such additives break down, problems of poor engine running, even stalling, and corrosion of system components (including biologically aggravated corrosion) arise. Stability problems have been found to be particularly acute with the higher fraction distillate hydrocarbon fuels (such as gasoline, kerosene and normal diesel fuels) in which separation of the oil and water components often occurs in a very short period of time. Where biocides are added to the emulsion to combat biochemically aggravated corrosion, this adds more complications to the already difficult task of attempting to formulate fuel additives for the stable mixing

of oil and water. This is because the biocides are treating the symptoms and not the underlying cause of the problems.

Our prior United Kingdom Patent No. 2217229 describes solubilising compositions that overcome these difficulties and allow oil and water, especially diesel oil and water, to be combined as a clear, stable homogenous solution. The solubilising compositions of that patent contain a minimum of three components and include a polyglycol ether of a higher fatty acid.

The present application provides novel solubilising compositions that have improved properties over prior art compositions, particularly in allowing solutions to be formed with the addition of markedly reduced amounts of additive. The present invention also allows effective compositions to be prepared from fewer components than those of our above-mentioned prior patent, with the possibility of simpler handling and reduced costs.

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The compositions of the present invention allow the formation of oil-water mixtures that are true solutions or behave as such. Any mixture that is clear, homogeneous and has substantially the same stability as a true solution is thus to be regarded as a "stable solution" for the purposes of the present invention.

The present invention accordingly provides a composition for combining oil and water as a stable solution which essentially comprises:

- 20 (a) an oil-soluble grade of alcohol ethoxylate having a hydroxyl number greater than 160 and melting point below -15°C; and
 - a complex mixture of higher fatty acid diethanolamides derived from coconut oil.
 or from its synthetic equivalent.

The composition may, if desired, comprise minor proportions of (c) ethylene glycol monobutyl ether or ethylene glycol monobutyl ester, or (d) an alcohol containing 5 or more carbon atoms. However, satisfactory results can be obtained in the absence of components (c) and (d), although the presence of component (c), or components (c) and (d) can be desirable in some circumstances. In particular, component (c) may enhance the solubility of the composition in the oil and water mixture, whereas component (d) may reduce the volume of composition required in order to produce a stable solution.

The complex mixture of higher fatty acid diethanolamides derived from coconut oil, or from its synthetic equivalent, is to be understood as being "complex" in the sense that it contains a plurality of different chemicals of the diethanolamide species, including

lauric, myristic, and oleic diethanolomides in significant quantities, generally together with other diethanolamides in less significant quantities, and generally a number of unidentified ingredients, possibly including such compounds as monoethanolamides and others that were present in the original biological source or were obtained as by-products during formation of the diethanaolamides. "Caflon CD" (Trade Mark) is a suitable commercially available mixture, largely composed of diethanolamides, derived from coconut oil.

The components of the composition are appropriately mixed together in amounts which would allow a stable solution to be formed on addition to the intended oil and water mixture.

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The present invention further provides a method of forming a stable solution of an oil and water mixture comprising adding to the oil and water mixture a composition of the present invention in an amount sufficient to provide a clear stable solution on mixing, and such a solution when so produced. Although the components could be added individually, it is contemplated that the composition would normally be prepared prior to the addition to the oil and water mixture, rather than being formed in situ.

The number of parts by volume of each component in the composition is selected so as to allow for the formation of a stable solution of the intended oil and water mixture. The exact amounts of each component of the composition required to give a solution of the intended oil-water combination may easily be found by simple experiment. Particularly improved performance over the compositions of our prior patent is found when treating 0.5 to 80% by volume of diesel oil in water. However the effective range of each formulation in treating diesel/water mixtures is reduced somewhat to avoid just an emulsion being formed instead of a cosmetically clear solution.

The range of water-contaminated diesel oil which may be treated by the compositions of the present invention comprising components (a), (b) and optionally (c) or (d) is 0 to 17% v/v water in diesel. This represents an increased ability to take water into clear "stable solution" as compared with compositions of our above-mentioned prior patent. Further said first compositions show improved "additive effectiveness" (i.e. a lower volume of composition added to the water diesel mixture will still produce a water/diesel "solution"). At 2.0% v/v water contamination the improvement is 12.5%, whereas at 10.0% v/v water contamination the improvement is 37% to 47%. By using different proportions of the components of the additive, it is possible to achieve long term stabilisation of diesel/ ethanol (20% ethanol v/v) mixtures from atmospheric deterioration

(i.e. with water contamination at <0.05% v/v). A more limited stability of ethanol/diesel solutions (20% ethanol v/v) of better than 14 days may be achieved in cases of severe water contamination (approximately 1% water v/v).

The components of the composition are all readily available commercially.

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Suitable alcohol ethoxylates of component (a) are for example "Neodol 91 2.5" (formerly available as "Dobanol 91/2.5") (Trade Marks of Shell Chemicals) and "Synperonic 91/2.5" or "Synperonic A 3" (Trade Marks of I.C.I. Plc.). Particularly preferred ethoxylates of component (a) are those based on C₉ and C₁₁ alcohol mixtures, more especially mixtures also including alcohols having chains with even numbers of carbon atoms.

"Neodol 91 2.5" is such an ethoxylate obtained by the addition of 2.5 moles of ethylene oxide per mole of Neodol 91 (a mixture of C₉, C₁₀ and C₁₁ alcohols).

The less highly preferred "Synperonic 91" range is manufactured by the base catalysed ethoxylation of Synprol 91 - a fully saturated synthetic primary alcohol which contains only C₉ and C₁₁ alkyl chains and typically consists of 50% w/w linear alcohols with the remainder being monobranched and predominantly the 2-methyl isomer.

As already mentioned, the compositions of the present invention show improved additive effectiveness compared with those of our above-mentioned prior patent.

As an example, for forming a solution of 0-5% v/v mixture of water in diesel, an appropriate composition would comprise approximately:

 $0.6 V_H \pm 2.2$ parts by volume of component (a),

1.3 $ln(V_H) + 1.2$ parts by volume of component (b), and

0 to 1 part by volume of components (c),

where V_H is the percentage by volume of water in diesel and "ln" means natural logarithm.

Preferred compositions according to the present invention for forming a solution of up to 2% v/v mixture of water in diesel comprise 1 to 2 parts by volume of component (b) per 100 parts by volume of diesel. A particularly preferred composition comprises about 1.5 parts by volume of component (a), 1 to 2 parts by volume of component (b), about 0.5 parts by volume of component (c), and about 1 part by volume of component (d)

Preferred compositions according to the present invention for forming a solution of up to from 2% to 4% v/v mixture of water in diesel comprise 2 to 3 parts by volume of component (b).

Preferred compositions according to the present invention for forming a solution of up to from 4% to 7% v/v mixture of water in diesel comprise 2.5 to 4 parts by volume of component (b). A particularly preferred composition comprises about 3 parts by volume of component (a), about 4 parts by volume of component (b), 0.5 parts by volume of component (c), and about 1 parts by volume of component (d).

Preferred compositions according to the present invention for forming a solution of up to from 7% to 10% v/v mixture of water in diesel comprise 5 to 6 parts by volume of component (b). A particularly preferred composition comprises about 8 parts by volume of component (a), about 6 parts by volume of component (b), 0.5 parts by volume of component (c), and about 1 parts by volume of component (d).

The composition of the present invention not only allows ready mixing of fuel oils and water but also allows the addition of further substances as fuel extenders in the oilwater solution. Thus, for example, animal and vegetable oils, even including tallow and butter, may be added successfully to diesel oil and water to form a clear solution on addition of the composition of this invention.

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Other extenders which contain water or are hygroscopic - such as alcohols (other than the alcohols of component (d)) - may also be successfully added to fuel oils to form permanent mixtures with the fuel oil. A number of previous attempts to extend fuel with alcohols, for example, have not been wholly successful because of phase separation and subsequent burning out of valves in the engine by the resulting pure alcohol component.

The compositions of the present invention are particularly effective in the stabilisation of ethanol/diesel mixtures.

Further, the compositions of the present invention are particularly effective in allowing the formation of stable solutions that show no sign of breaking down after extended periods of time, in contrast with the emulsions formed with prior art additives. Thus oil and water mixtures incorporating compositions of the invention have formed clear solutions showing no sign of separation of the components over periods of as long as three years or more.

The uses of solubilising compositions are by no means restricted to these uses but include numerous other applications such as oil recovery, the safe cleansing under pressure of oil tanks (conventional low flash point solvents being unnecessary) and as additives to jet fuel. However yet further applications will readily occur to the man skilled in the art.

The following examples illustrate the formation of compositions of the invention and their use for the formation of stable solutions of water and oil.

Example 1

A series of tests were carried out to determine the optimum content of Caflon (as component (b) of the present invention) in a solubilising mixture of the present invention.

A simple three component system was used in which a fixed quantity of Egme (0.5 ml/100 ml fuel) and a predetermined amount of Caflon was added to a known amount of diesel and water. The mixture was titrated with Neodol 91-2.5 (as component (a) of the present invention) to afford clear liquid solutions. The results (shown in Figure 1) suggested that certain formulations would lead to a minimum additive requirement (optimum result). It would appear that certain points on the 4% and 8% water content curves of Figure 1 are anomalous in this particular experiment, however, an estimate of the optimum Caflon content ("Cf-opt") can be established from the minimum values on the respective curves.

A relationship between increasing water content and increasing the optimum Caflon content was observed. Within the scope of experimental variation and error, it would appear that the relationship was approximately linear for the range 1-5% water and with higher water content, such deviations from linearity would be small. Similarly it was possible to relate the amount of the primary component. Neodol 91-2.5, required at the optimum Caflon content to the water content, as shown in the following Table 1 in which, V_H is the percentage by volume of water in diesel. V_{Cf} is the percentage by volume of Caflon in diesel and V_{N1} is the percentage by volume of Neodol in diesel

V _H	V _{Cf}	V _{NI}	$(V_{NI} + V_{Cf})$	ln (V _H)
	1.41	2.59	4.0	0.0000
2	2.06	3.58	5.64	0.6931
3	2.53	4.24	6.77	1.0986
4	3.28	5.36	8.64	1.6094
5	3.5	5.64	9.77	2.0794
8	3.17	1 0.0		Table 1.

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In this instance, the quantity of Caflon and Neodol required may be expressed by the following equations:

 $V_{Cf} = 1.2132 \ln (V_H) + 1.3470$ (correlation coefficient = 0.9817), and $V_{N1} = 1.7841 \ln{(V_H)} + 2.2533$

Example 2

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1ml water was mixed with 100ml diesel and 0.5 ml Egme and the amount of Caflon indicated in the following table were added. Neodol 91-2.5 was titrated into the mixture with agitation and the amount needed to produce a clear solution was noted. The results of three trials are indicated in the following table.

Total additive volumes						
Caflon (ml)	Trial (a)	Trial (b)	Trial(c)	Average (standard)		
		5.90	5.95	5.95		
0.5	5.15	4.90	4.80	4.95		
1.0		5.15	5.10	5.15		
2.0	5.20	6.30	6.30	6.20		
3.0	6.00		-	7.10		
4.0	7.10	7.05		7.70		
5.0	7.40	8.00		Table 2.		

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Example 3

The contribution of ethylene glycol monobutyl ether/ester ("Egme" - component (c)) to compositions of the present invention was evaluated by a series of experiments in which titrations were carried out to determine the relationship between total additive content and the amount of Egme present for a fixed amount of water in diesel using Neodol 91-2.5 as the primary component. The results indicated that Egme did not reduce the level of Neodol required. However, it was observed that titrations which contained Egme were easier to perform. This was because of a reduction in the formation of waxy scales, sometimes observed when using Neodol 91-2.5 or Caflon alone.

Although the wax-like scales/globules (intermediate stage) did not seem to change the overall volume of additive required, they resulted in increased difficulty and hence increased time for blending before producing the clear fuel. This could lead to a possible problem of a temporary blockage in fuel pipes caused by these wax-like globules in some circumstances, although the wax-like globules would eventually dissolve. If a formulation comprising just Neodol 91-2.5 and Caflon is used, care must therefore be taken to ensure that complete solution of the formulation in the fuel has occurred before using the fuel.

In preferred compositions Egme should be included, but in a very small proportion -e.g. in an amount of not more than 0.5 parts by volume/100 parts by volume of fuel.

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Example 4

The contribution of component (d)) to compositions of the present invention was likewise evaluated by a series of experiments in which titrations were carried out to determine the relationship between total additive content and the amount of various alcohols present for a fixed amount of water in diesel, using a composition similar to the composition of Example 2 as the primary component. The results in the following Table 3 and the chart of Figure 2 show the total volume of additive that was required to produce a clear solution on addition to diesel with a 1% by volume water content:

Total additive volumes							
Caflon	0.5	1.0	2.0	3.0	4.0	5.0	Reference in chart
Component Standard* Methyl alcohol Ethyl alcohol Propyl alcohol t-Butyl alcohol n-Butyl alcohol n-Pentyl alcohol n-Hexyl alcohol n-Octyl alcohol n-Nonyl alcohol n-Decyl alcohol n-Decyl alcohol n-Decyl alcohol n-Undecyl alcohol	5.7 8.7 7.55 6.75 7.75 6.1 5.7 6.1 5.6 5.65 5.65	4.3 8.1 7.2 6.2 6.1 5.0 3.95 3.35 3.3 2.9 2.85 2.85 2.85	4.2 8.3 7.75 6.3 6.0 5.3 5.0 4.25 4.25 4.2 4.2 4.3 4.2	4.8 8.8 8.05 7.0 6.8 6.1 5.65	9.8 8.9 7.9 7.5 7.95	10.5 9.7	sl a b c d e f g h i j k

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In each instance the formulation comprised the volume of Caflon in ml. as indicated in the column heading, 0.5 ml of Egme, 1ml of the indicated alcohol and the balance of Neodol.

^{*&}quot;Standard" means the content of Neodol and Egme in a formulation of Caflon. Neodol and Egme.

It is clear that the critical size is approximately the size of the hydrophobic group in propyl alcohol, below which the alcohols have a negative effect on the additive. It should be noted that the amount of Caflon, as far as it possible to determine, is again close to the optimum expected value. It will be seen that, at certain additive volumes, alcohols containing five or more carbon atoms reduce the amount of additive required to solubilise water-contaminated diesel.

Of the higher alcohols, hexyl alcohol is preferred as it is normally available at the least expense.

Example 5 10

A preferred composition was prepared by adding together the following components in the stated proportions by volume:

90 parts Neodol 91 2.5

75 parts Caflon CD

12.5 parts EGME. 15

The components were mixed vigorously together to form a homogeneous solution.

Example 6

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The effect of the use of the composition of Example 5 in diesel fuel on engine 20 performance was tested by (A) on-road fuel consumption testing, and (B) dynamometer fuel testing.

A Toyota Corolla 1.7 litre diesel station wagon that had travelled 129,000 kilometres was used as the test vehicle. A compression test on this vehicle's engine showed the following results - cylinder no.1 - 370 lb/sq.in, cylinder no.2 - 355 lb/sq.in, cylinder no.3 - 360 lb/sq.in, cylinder no.4 - 370 lb/sq.in. These are average compression rates for this model's engine at these kilometres, and are reasonably even. An optimum would be approximately 420 lb/sq.in for all cylinders on a newly run-in vehicle.

A high kilometre vehicle was used to ensure running-in variables were avoided and show if any advantages were to be gained through cleaning effects on the injectors. pumps, combustion chambers etc.

On-road fuel consumption testing.

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Treated fuel was prepared by adding 1 ml of the composition of Example 5 to each litre of diesel fuel. The kilometres travelled, fuel used and speedometer readings 5 were recorded in a logbook and readings were taken from vehicle speedometer and certified fuel pump meters. Different people drove the vehicle daily in different applications, thus minimising the potential of driver awareness effecting the test results. The results of this test are shown on Figure 3, in which the hatched area represents the results for the treated fuel and the unhatched areas show the results before and after treatment.

The average fuel consumption before treatment was 42.58 mpg (15.07 kilometres/litre), whereas the average fuel consumption during treatment was 46.70 mpg (16.53 kilometres/litre) - a 9% improvement, and the average fuel consumption after treatment had stopped was 44.61 mpg (15.78 kilometres/litre) - a 4.5% reduction on the results during treatment. (The normal margin of error in testing here might have been expected to be \pm 3%.) Temperature variations during the course of the study may have had a slight effect on the results. However the pre-treatment test was carried out in early Spring, whereas the bulk of the test was carried out over the dry summer period. The expected reduction in performance under these circumstances could, in fact, have reduced the improvement shown in using the treated fuel.

In any event, these figures obtained would equate to a good percentage saving in fuel consumption and would certainly be more than enough to cover the costs of treatment.

Dynamometer fuel testing. 25 B.

The effect of using treated diesel fuel, again with the same content of the composition of Example 5, was tested using a water driven dynamometer under fine weather conditions with low humidity.

The test involved running a warm engine at full throttle loaded to 100 kph with a set quantity of fuel. A full throttle setting and governed engine speed using a dynamometer were employed to try to alleviate driver error. This appeared to have worked in view of the consistency of results that were obtained. Temperature and humidity variations during the course of this test may have had slight effects, given the interval between tests. Humidity readings were taken at the time and were very close at 68% and 65 %, respectively.

The results are shown in the following Table 4.

Pre- testing with composition of E	diesel containing the xample 5	After testing with diesel containing the composition of Example 5		
1st run 2nd run 3rd run	2.7 km/litre 2.6 km/litre 2.7 km/litre	1st run 2nd run 3rd run	2.9 km/litre 2.9 km/litre 2.9 km/litre Table 4.	

Just under 9% improvement on the dynamometer tests were achieved after using diesel containing the composition of Example 5 for 27,000 kilometres. This was very close to the results that were obtained on road testing.

It might have been expected that a greater improvement would have been shown in the dynamometer test than in the on-road test, as the road testing took into account the improvement period of approximately 5,000 kilometres. That this improvement was not shown, is perhaps due of the extra stress that was incurred when running a diesel engine at full throttle and to the fact that the injector pump is designed to over-fuel under these conditions.

It will be noted from Figure 3 that, when the use of diesel containing the composition of Example 5 was started there was a period of gradual improvement in performance, and when the use was stopped the fuel consumption rate did not immediately return to its original higher value, but stayed noticeably better. This may be the result of the cleaning effects of extended use of the composition of Example 5. As the composition of Example 5 has a neutral effect on the octane and cetane ratings of diesel, the direct improvement from its use could be explained by its strong surfactant properties helping to optimise the atomisation of the fuel and thus giving a cleaner more complete fuel burn.

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It will be understood that the invention has been described above purely by way of example, and that various modifications of detail can be made within the ambit of the invention.

CLAIMS

- A composition for combining fuel oil and water as a stable solution which essentially comprises
- a) an oil-soluble grade of alcohol ethoxylate having a hydroxyl number greater than 160 and melting point below -15°C, and
 - (b) a complex mixture of higher fatty acid diethanolamides derived from coconut oil, or from its synthetic equivalent.
- 10 2. A composition according to claim 1, which further comprises
 - (c) ethylene glycol monobutyl ether or ethylene glycol monobutyl ester.
 - 3. A composition according to claim 1 or 2, which further comprises
 - (d) an alcohol containing 5 or more carbon atoms.

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- 4. A composition according to claim 1 for forming a solution of 0-5% v/v mixture of water in diesel comprising approximately:
 - $0.6\ V_H$ + 2.2 parts by volume of component (a) ,
 - $1.3 \ln{(V_H)} + 1.2$ parts by volume of component (b), and
- 20 0 to 1 part by volume of components (c),

where $V_{\rm H}$ is the percentage by volume of water in diesel and "ln" means natural logarithm.

- 5. A composition according to any of claims 1 to 3 for forming a solution of up to 2% v/v mixture of water in diesel comprising about 1.5 parts by volume of component (a), 1 to 2 parts by volume of component (b), about 0.5 parts by volume of component (c), and about 1 part by volume of component (d).
- 6. A composition according to any of claims 1 to 3 for forming a solution of from 4 to 7% v/v mixture of water in diesel comprising about 3 parts by volume of component (a), about 4 parts by volume of component (b), 0.5 parts by volume of component (c), and about 1 part by volume of component (d).

7. A composition according to any of claims 1 to 3 for forming a solution of from 7% to 10% v/v mixture of water in diesel comprising about 8 parts by volume of component (a), about 6 parts by volume of component (b), 0.5 parts by volume of component (c), and about 1 part by volume of component (d).

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- 8. A method of forming a solution of a mixture of oil and water comprising adding a composition according to any of claims 1 to 7, to the oil and water mixture in an amount sufficient to provide a clear solution on mixing.
- 10 9. A solution whenever produced by the process of claim 8.
 - 10. A composition according to claim 1, substantially as described in any one of the Examples.
- 15 11. A solution according to any of claims 1 to 7 and 9, additionally containing a fuel extender.
 - 12. A solution according to claim 11, in which the fuel extender is tallow, butter, a vegetable oil or a C_1 C_4 alcohol.

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13. A solution according to claim 12, in which the fuel extender is ethanol.





Application No: Claims searched: GB 9807792.8

All

Examiner: Date of search: Michael R. Wendt

24 July 1998

Patents Act 1977 Search Report under Section 17

Databases searched:

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

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Documents considered to be relevant:

Docum	ents considered to be	e relevant:	Relevant
Category	Identity of document and relevant passage		
X	GB 2217229 A	(ENERSOLVE) - referred to in the application - see whole document.	1, 2, 8 & 11 at least.
X,E	WO 98/17745 A1	(HAZEL) e.g. see Claim 1. Page 1 at paragraph 3. Example 1.	1 & 8
A	Chemical Abstract Abstract.	No. 95:13 5609 & D E 2940782 A (HENKEL). See	

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